

# PATENT SPECIFICATION

DRAWINGS ATTACHED

1.031.764

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Date of Application and filing Complete Specification March 15, 1963.  
No. 10398/63.

Application made in Germany (No. D38532 IVa/12g) on March 30, 1962.  
Complete Specification Published June 2, 1966.

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Index at acceptance: —CI A(13, NX2)

Int. Cl.: —C 01 b 13/14, C 01 b 33/18

## COMPLETE SPECIFICATION

### Improvements in or relating to the Surface Modification of Highly Disposed Oxides

We DEUTSCHE GOLD-UND SILBER-SCHEID-ANSTALT VORMALS ROESSLER, of 9 Weiss-frauenstrasse, Frankfurt (Main), Germany, a body corporate organised under the laws of Germany, do hereby declare that the following is a full and complete specification of the improvements in or relating to the surface modification of highly disposed oxides, which have been prepared separately to form oxide mixtures.

The use of halogen-containing starting

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## ERRATUM

SPECIFICATION No. 1,031,764  
Amendment No. 1

Page 1, Title, for "Highly Disposed Oxides"  
read "Highly Dispersed Oxides"

THE PATENT OFFICE  
5th August 1966

condensable gaseous reaction products. The water-vapour-forming gas mixtures employed may consist of combustible, in particular hydrogen-containing or hydrogen-yielding gases, and of incombustible, oxygen-containing gases. Oxides of this type are obtained with a particle size of less than 150 microns.

Examples of suitable starting materials are volatile halides, the chlorides or fluorides being particularly suitable. It is also possible to start with several metals or metalloids or their volatile compounds simultaneously, and to react these together, so that the oxides precipitate in the form of mixed oxides. It

dered hydrophobic by treatment with silicone oils. However, to effect this, the dry, powdery silica must be suspended in an organic liquid.

Lastly, metal oxides obtained by pyrogenous processes and which have free hydroxyl groups on their surface have previously been treated in countercurrent with gaseous or easily evaporable substances which can react with the hydroxyl groups, forming ethers, esters or acetals. Examples of this class of substances are formaldehyde and ketene. This treatment has also been effected following previous or simultaneous hydrolysis with water or steam. However, hydropho-

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## COMPLETE SPECIFICATION

### Improvements in or relating to the Surface Modification of Highly Dispersed Oxides

We DEUTSCHE GOLD-UND SILBER-SCHEI-  
DEANSTALT VORMALS ROESSLER, of 9 Weiss-  
frauenstrasse, Frankfurt (Main), Germany, a  
body corporate organised under the laws  
of Germany, do hereby declare the inven-  
tion, for which we pray that a patent may be  
granted to us, and the method by which it  
is to be performed, to be particularly de-  
scribed in and by the following statement:—  
This invention relates to a process where-  
by it is possible to alter permanently the  
properties of highly dispersed pyrogenically  
produced oxides, oxide mixtures, and mixed  
oxides of metals and/or silicon, by conver-  
sion of the hydroxyl groups located on their  
surfaces.  
It is known to produce highly dispersed  
oxides by the reaction of metals or silicon or  
their volatile compounds in the vapour phase  
at high temperature in the presence of, for  
example, water or oxygen. For example, the  
volatile metal or metalloids halides can be  
reacted in the gas phase with water vapour  
or water vapour-yielding gas mixtures, and  
the oxides, produced in the form of their  
aerosols, are subsequently isolated at tem-  
peratures above the dew points of the easily  
condensable gaseous reaction products. The  
water-vapour-forming gas mixtures employed  
may consist of combustible, in particular  
hydrogen-containing or hydrogen-yielding  
gases, and of incombustible, oxygen-contain-  
ing gases. Oxides of this type are obtained  
with a particle size of less than 150 milli-  
microns.  
Examples of suitable starting materials are  
volatile halides, the chlorides or fluorides be-  
ing particularly suitable. It is also possible  
to start with several metals or metalloids or  
their volatile compounds simultaneously, and  
to react these together, so that the oxides  
precipitate in the form of mixed oxides. It

is also possible to combine various oxides  
which have been prepared separately to form  
oxide mixtures.

The use of halogen-containing starting  
materials in the the vapour phase oxidation or  
hydrolysis reaction, for example, the use of  
silicon tetrachloride or silicon tetrafluoride,  
leads to the formation of products which,  
owing to their high adsorptive capacity, con-  
tain large amounts of hydrogen halide as well  
as halogen bound directly to the metal-  
or silicon atom. Oxides of this type have a  
strongly acid reaction. Their halogen acid con-  
tent may be for example, 0.1%, so that a  
pH of about 1.8 results (the pH of such  
oxides is obtained as explained below). Oxides  
of this type may display hydrophilic proper-  
ties.

It is further known to render powdery silica  
hydrophobic by treating it with alkylchloro-  
silanes. Here, the chlorosilane reacts with the  
water adsorbed on the surface of the silica,  
forming hydrochloric acid. The silica ren-  
dered hydrophobic in this manner must be  
subsequently freed from the hydrochloric acid  
thus formed.

Further, powdery silica has also been ren-  
dered hydrophobic by treatment with silicone  
oils. However, to effect this, the dry,  
powdery silica must be suspended in an  
organic liquid.

Lastly, metal oxides obtained by pyro-  
genous processes and which have free hydroxyl  
groups on their surface have previously been  
treated in countercurrent with gaseous or  
easily evaporable substances which can react  
with the hydroxyl groups, forming ethers,  
esters or acetals. Examples of this class  
of substances are formaldehyde and ketene.  
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lowing previous or simultaneous hydrolysis  
with water or steam. However, hydropho-

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bic oxides prepared in this way could only rarely be obtained in a stable form, since in fact no chemical reaction occurred with the hydroxyl groups of the surface, but merely the superficially adsorbed water was reacted, whereas a stable hydrophobisation occurs only when it is brought about by actual chemical reaction with the hydroxyl groups. Thus, for example, only highly dispersed oxides, which have been hydrophobised by chemical reaction, cannot be shaken out from carbon tetrachloride with water. Other products, in which the hydroxyl groups have not been reacted chemically, go into the aqueous phase during this process, because the carbon tetrachloride merely detaches the adsorbed organic molecules from the surface. Similar conditions prevail in reactions with other substances which are not hydrophobising agents and where, insofar as these substances are capable of reacting with water, reaction with the hydroxyl groups is prevented by the presence of adsorbed water or adsorbed hydrogen halide.

It has now been found that finely divided oxides, oxide mixtures, or mixed oxides of metals and/or silicon obtained by the reaction of volatile compounds of these metals and/or silicon in the vapour phase in the presence of gases or vapours having a hydrolysing and/or oxidising effect, can be permanently modified in their properties by reacting the free or liberated hydroxyl groups located on the surfaces of the freshly prepared oxides with a vapour substance (A), which is capable of reaction with hydroxyl groups, such oxides being held in by, and in parallel current to the substance A, by mixing these oxides, oxide-mixtures or mixed oxides after substantially freeing them from halogen, hydrogen halide and adsorbed water, as homogeneously as possible with the substance (A) in the absence of oxygen, and feeding this mixture continuously, together with small amounts of steam and, possibly, an inert gas, to a vertical kiln in which the solid particles are heated to temperatures from 200 to 800°C., preferably from 400 to 600°C., separating the solid and gaseous reaction products from each other, if necessary drying, and if desired de-acidifying, the solid product, and avoiding contact with oxygen before the product cools down to temperatures below 200°C.

The particle size in the oxide suspension naturally depends upon the nature of the kiln used and upon the flow velocities used. In the present specification, oxide suspensions are formed by, for example, silica particles at a flow velocity of from 2 to 10 cm/sec.

As stated above, the oxides which have been deacidified to the greatest possible extent, i.e., which have been freed of halogen or hydrogen halides deriving from their preparation are particularly suitable for treatment by the process of the invention. In contrast to the original products, these oxides display a pH of about 4.0. The pH value of the oxides are measured, by known methods, using a glass electrode in a disposition of 4 g. of silica dioxide in 100 ml. of water. The de-acidification can be effected by known processes, for example, by treatment with steam in a rotary kiln, on an endless steel belt or in screw conveyors at elevated temperatures. However, the de-acidification can be carried out in a particularly advantageous manner as described in our co-pending Application No. 15079/62 (Serial No. 1003957) whereby deacidification is carried out within a treatment chamber in a rotating suspension in an ascending steam current, which may be diluted with an inert gas for the purpose of regulating the partial pressure of the steam, at temperatures from 450°C to 800°C, with continuous removal of the impurity-bearing gases. In the context of the specification, oxides which contain less than 0.5 weight % of adsorbed hydrogen halide, calculated on the oxide, are considered as being substantially de-acidified. Preferably, the hydrogen halide content is about 0.1%.

Furthermore, the oxides to be treated according to the process of the invention must be substantially free of water, i.e. they should contain a less than monomolecular water layer which, for a specific surface area of 200 m<sup>2</sup>/g, corresponds to less than 0.9% water. This condition is automatically achieved in the course of a thoroughgoing deacidification, particularly when the latter is carried out according to the process of our co-pending Application No. 15079/62 (Serial No. 1003957). Otherwise, any known drying methods may be used. It is particularly advantageous to put the process of the invention into effect directly following the preparation of the oxides to be treated, because the adsorbed water content is very low at this stage. Thus, for example, the treatment according to the invention may directly follow the cyclone separation.

It has been found particularly advantageous, that the deacidification and the surface treatment do not have to be necessarily carried out in separate steps. The reaction conditions of the surface treatment are so calculated that deacidification and dehydration can take place simultaneously. It is also possible to effect a subsequent deacidification.

The amounts of steam, in the presence of which the surface treatment with the substance or substances (A) is carried out, must be small, otherwise the thermally decomposed hydroxyl groups will be reconstituted. It is advisable to employ from 0.5 to 2.0 mols water for 100 m<sup>2</sup> of oxide surface. Preferably, a gas is employed for this purpose which contains from about 10 to 100 g water/m<sup>3</sup>.

The amounts of the substances (A) to be reacted should be calculated as a function

of the surface area and the purpose of the treatment. Thus, for example, a highly dispersed silica with a surface area of 200 m<sup>2</sup>/g has 1 m mol/g of free hydroxyl groups; consequently, the theoretical amount of substance (A) to be employed is 1 m mol/g. In practice however it is advisable to employ 1.5 m mol/g.

The steam used can be diluted, preferably with an inert gas such as nitrogen. This allows the regulation of the partial pressure of the steam within the treatment chamber. Its setting may be varied within wide limits and depends only on the requirements for the preparation of products with predetermined characteristics. In general, it is advisable not to use saturated steam.

The reaction products must be separated from the gaseous products such as hydrogen halides and excess treating agents, before the drying step. It is essential for the successful working of the process, that these gases are continuously evacuated, i.e., that they are separated from the solid particles before the oxides have cooled down to a temperature at which an appreciable re-adsorption could take place. If the product treated has organic components, then it is necessary—in order to avoid combustion—to allow the entry of air or oxygen only when the temperature has dropped below 200°C.

In place of nitrogen, any inert gases can be used in which the surface modifying substance is incombustible, and which will not react with the hydroxyl groups. In addition, these gases must remain stable at the temperature of the reaction. To this class belong for example, the inert gases (neon, xenon, argon . . .), hydrocarbons and chlorinated hydrocarbons.

The substances (A) which can be employed for the surface treatment include all compounds which can react with the hydroxyl groups, for example, by etherification, esterification or ketal-formation. Examples of such substances are alcohols, aldehydes, ketones and alkylene oxides. It is particularly advantageous to react the oxides with the corresponding halides of the oxides to be treated. For example, silica can be treated with silicon tetrachloride according to the process of the invention, so that stable, cross-linked agglomerates of the oxides are obtained, which are characterised by an increased flattening-effect.

In some cases, it may be desirable to obtain an oxide with a pH greater than 4, the value normally obtained after treating the oxide with hydrophobising agents. These products can be obtained by initially reacting the oxide with alkaline compounds, preferably alcoholic alkaline compounds, especially alcoholic potassium hydroxide.

Further examples of substances (A) which can be used are, in particular alkyl- or aryl-

or mixed alkyl-aryl-halogeno-silanes. In addition to the preferred dimethyl-dichlorosilane, other examples of such substances are: ethyltrichloro-silane, amyltrichlorosilane, vinyltrichlorosilane, phenyltrichlorosilane, methyltrichlorosilane, methyldichlorosilane, methylvinyl-dichlorosilane, trimethylchlorosilane, diphenyldichlorosilane, bis-trichlorosilyl-ethane, bis-trichlorosilylbenzene.

Silane esters can be also used, such as ethyltriethoxy silane, vinyl-triethoxy-silane, phenyl-triethoxy-silane, amyl-triethoxy-silane, dimethyl-diethoxysilane, diphenyl-diethoxysilane,  $\beta$ -carbethoxyethyl-triethoxy-silane and  $\beta$ -carbethoxypropylmethyldiethoxy-silane. The last named group is not, however, counted among the preferred substances, since they have less stable hydrophobic properties. They have nevertheless the advantage that no hydrogen halide is formed during the corresponding reaction so that subsequent de-acidification is unnecessary.

Advantageously, those agents are chosen for the treatment which can be easily vaporised. It is a particular advantage, that all the stages of the process according to the invention can be carried out simultaneously in a single treatment chamber, that is to say, all operations, such as de-acidifications, water-removal, charging with the treating agent, reaction with this agent, separation of the gaseous reaction products as well as the drying are carried out in a single operational step.

It is also of particular importance that the products obtainable by the process according to the invention retain their ability to thicken organic liquids. Thus, for example, a silica which has been rendered hydrophobic according to the process of the invention gives a gel with about 7% weight volume of paraffin oil or carbon tetrachloride. With an untreated silica, this effect is also obtained with 6 to 7% of the above liquids. In contrast hereto, products hydrophobised by other processes display an insufficient thickening ability.

The preferred form of the parallel current process is carried out by conducting the entire treatment in a treatment chamber which is so dimensioned that the suspension can progress upwards continuously to leave the chamber at its upper portion, preferably after having traversed a settling zone. The exhaust gases are continuously withdrawn at the upper end of the treatment chamber; the hydrogen halides or oxide particles therein contained can be recycled to another batch. A settling zone can be created simply by enlarging the furnace cross-section.

The oxides are fed into the treatment chamber preferably together with the inert gas and/or steam and are maintained in the spiral path until they leave the chamber. The oxides are thus in motion relative to

the steam. The oxides progress steadily towards the upper outlet opening. For such a process it is essential that the oxides to be treated have a fluidisable particle size. It was thus entirely surprising that such a fluidisation and a subsequent separation of the solid particles from the carrier medium could be executed when employing highly dispersed oxides having a particle size of less than 150 millimicrons. In particular, it was surprising that a parallel current system could be used. Nor could it be foreseen that a successful treatment could be carried out with a very brief reaction time.

The velocity of the particles flowing through the treatment chamber must be at least high enough to fulfil the fluidisation requirements. When highly dispersed oxides are treated this is achieved when the flow velocity of the carrier medium reaches about 2.0 cm/sec. The velocity can be determined in a simple manner by giving the treatment chamber suitable dimensions and/or regulating the steam—or gas volume.

The residence time of the oxides in the treatment chamber can be varied by altering the dimensions of the chamber. It is equally possible to connect several treatment chambers in series.

It is of importance for the successful treatment of the oxides or oxide mixtures or mixed oxides to bring the suspended particles to the above indicated temperatures. How long they are kept at these temperatures depends on the desired properties of the finished products. The treatment is completed in a few minutes where high-powered heating devices are used. If, on the contrary, heating devices with lower output are employed, then the residence time of the particles must be extended correspondingly. Experiments have shown that an efficient heating of the treatment chamber cannot in general be achieved by external heating, particularly when the process has to be carried out on an industrial scale. However, internal heating always leads to successful results besides being economically advantageous, especially so, if the incoming input gases are heated. The internal heating may be provided by means of known devices, such as infra-red heaters, high-frequency fields or by a flame jet directed into the interior of the chamber. However, by reason of their high efficiency and their resistance to the reaction medium, electric heating elements are preferred for internal heating, arranged within an insulating material, for example, quartz tubes. Particularly good results were obtained with members in the shape of blades, fins or vanes which can be easily arranged in considerable numbers within the treatment chamber.

As mentioned above, it is also advantageous to employ a hot, inert gas for the treatment according to the invention, in such a man-

ner, that the gas provides at least a part of the heat required for the treatment. The inert gas is introduced into the treatment chamber expediently together with the steam. This can be effected, for example, by the injection of water into a heater with the help of a proportioning pump.

In a particularly advantageous variant of the process, the generation of steam and heat are coupled. This can be effected, for example, by burning one or more oxyhydrogen flames.

If in the process according to the invention an inert gas is also used, then this gas can be simultaneously used for the pneumatic conveying of the oxides to be treated and of the treating agents, by introducing them into the treatment chamber from below and admixing the steam at the same time. It has been surprisingly found that it is possible to operate with relatively small amounts of gas so that a low heat-output suffices.

Another advantageous feature of the process according to the invention is the fact that a complete and permanent reaction, for example, hydrophobising can be achieved with low expenditure of energy and that devices can be used for this purpose which do not contain moving parts. For this reason, ceramic construction materials can be employed as well as metallic materials of construction, which ensures a greater purity of the products.

A device which proved to be particularly advantageous for the execution of the process is illustrated in the accompanying drawing.

The device consists essentially of a vertical, tubular furnace 1 with feed elements 5 for the oxides, steam and the treating agent and a funnel-shaped enlargement 2 at its upper end which serves as a settling zone, together with an outlet 3 for the oxides treated and another outlet 4 for the waste gases. An additional gas heating arrangement 7 can be provided on the furnace. At the lowermost extremity of the reaction tube there may be provided a device 13 for the evacuation of grits.

The inert gas, for example, nitrogen, is introduced through pipes 8 and possibly through heater 9. The required amount of water is supplied through a conduit 10. The inert gas and the steam enter the furnace through a conduit 11. Simultaneously, oxide is fed into conduit 11 from the storage bins or from separators 12 which are connected in series to the oxide producing plant, which can be effected either by means of a bucket-wheel sluice or by gravity-conveying. In addition, the treating agent is introduced into the conduit 11 by means of an inert gas stream. The mixture ascends in the tubular portion of the furnace 1 where all reactions take place under the influence of the high

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temperatures. The oxide thus treated passes from the settling zone 2 into the outlet 3. Advantageously, a cyclone may be arranged above the settling zone. The reaction chamber is of course not restricted to tubular shape; other shapes, for example, conical, are also possible. The reaction chamber is preferably insulated against heat loss. A particular arrangement for the internal heating has given especially good results. This consists of rod-shaped or tubular, preferably electrically powered, heating elements 6, which may be arranged in the interior of the furnace, about perpendicular to its longitudinal axis. Preferably these heating elements may be so arranged, that their longitudinal axes are at an angle of 60° relative to each other.

It is known from U.S. Patent No. 2,993,809 to treat highly dispersed oxides with silanes in the vapour phase to render them hydrophobic. In this known process, however, the hydrophobising agent is added immediately after the formation of the oxides from the halides in the presence of steam and oxygen at temperatures of below 500°C, i.e. the hydrophobising agent is fed in at the burner nozzle itself. For this reason, precautions must be taken to avoid an excess of oxygen in the burner nozzle. Thus, the combustion must be carried out in a closed space and an inert gas would be used instead of the secondary air. In this case, however, the volume of the inert gas would be 30 times greater than in the process of the present invention. In addition, the hydrophobising is effected in this known process in the presence of the hydrogen halide liberated during the formation of the oxide, so that the hydrogen halide is present in high concentrations. The products obtainable by this known process therefore display a pH which does not exceed 2.0. Further, it is not possible to control the temperature in the reaction space. The excess of water required in the flame hydrolysis restricts the control of the desired particle size. Since the reaction proceeds uncontrolled, the residence time for the hydrophobising reaction cannot be regulated and the yield is correspondingly unfavourable.

The invention is further illustrated by the following Examples.

#### EXAMPLE 1.

4.5 kg. per hour of silica of pH 1.8 are fed into an apparatus which corresponds to the one illustrated in the Figure, having a fluid bed volume of 100 litres, together with 6 m<sup>3</sup>/hour nitrogen and 200 g/hour steam with the help of a carrier gas. The carrier gas consists of nitrogen and its flow is adjusted to 1 m<sup>3</sup>/hour. The charge is reacted with 720 g/m<sup>3</sup> dimethyldichlorosilane. A temperature of from 400 to 500°C is maintained in the interior of the furnace, by means of internal electrical heating. The flow rate

of the suspension is from 7 to 8 cm/sec.; the total residence time is from 18 to 20 minutes. After leaving the furnace, the silica displays a pH of 3.8. The product is only slightly altered in comparison with the starting material. The BET-surface is 310 m<sup>2</sup>/g, its loose density is 30 g/l and its tamped volume 22.3 g/l. The product has little tendency to cake.

#### EXAMPLE 2.

A silica obtained by flame hydrolysis of silicon tetrafluoride is deacidified in a fluidised bed at 800°C according to the process of our co-pending Application No. 15079/62 (Serial No. 1003957). This silica is reacted in a second fluidised bed connected in cascade with dimethyldichlorosilane in the manner described in Example 1. The product has the following properties.

loose density: 32 g/l  
tamped volume: 23.5 g/l  
pH: 3.7.

#### EXAMPLE 3.

A highly dispersed silica having a pH of 4.0 is treated, as in Example 1, in a fluidised bed heated to 200°C with a 10% alcoholic potassium hydroxide solution conveyed by 7 m<sup>3</sup>/hour nitrogen at a flow velocity of 3 to 5 cm/sec. The potassium hydroxide is introduced into the injector nitrogen stream by means of an atomiser nozzle at a rate of 0.8 m<sup>3</sup>/hour. The product thus obtained has a pH of 8.3; its potassium content is 1.03%, its ignition loss 1.03% and it has a surface area of 200 m<sup>2</sup>/g. Gel-formation occurs with 8.6 g of this product in 100 ml water, while 15.4 are required of the untreated product to effect this. In butanol, for example, a gel is formed with 5.8 g/100 ml, whereas 19.7 g of the untreated product are required to effect gel-formation. To form a gel in carbon tetrachloride, 5.6 g. of the treated product are required and 6.2 g of the untreated product to effect gel-formation.

#### EXAMPLE 4.

The process is carried out as in Example 1, but the temperature of the fluidised bed is from 600 to 800°C and from 6 to 8 m<sup>3</sup>/hour injector-nitrogen, 1 kg/hour water, 4 kg/hour highly dispersed silica and 400 g silicon tetrachloride are fed in, the latter corresponding to 10 weight%. The flow velocity is from 4 to 8 cm/sec, the residence time is a total from 2 to 5 minutes. A cross-linking as well as a de-acidification takes place during this period. The product has a pH of about 4.0 and is distinguished by a particularly stable agglomeration. Specification No. 796,646 describes and claims a process for the surface treatment of oxide material consisting of one or more finely divided inorganic oxides containing free OH

groups on their surface, wherein the oxide material is treated in substantially or completely anhydrous form in the absence of a liquid phase, preferably in a fluidised bed, with an alcohol which is in the vapour state.

# WHAT WE CLAIM IS:—

1. A continuous process for the production of hydrophobic oxides of metals and/or silicon by the surface treatment of freshly prepared finely divided oxides, oxide mixtures or mixed oxides of metals and/or silicon, obtained by the thermal decomposition of volatile compounds of the metals or of silicon in the vapour phase in the presence of gases or vapours having a hydrolysing or oxidising action, which oxides have hydroxyl groups located on their surfaces, wherein the oxides are freed from halogen, hydrogen halide and adsorbed water and are then mixed as homogeneously as possible, and with the exclusion of oxygen, with substances (A) capable of reaction with the surface hydroxyl groups, the mixture then being fed continuously, together with small amounts of steam, to a vertical treatment chamber in which the solid particles and the substance (A) are in parallel flow, the solid particles being in suspension, and are heated to a temperature of from 200° to 800°C., the solid and gaseous products there being separated from each other and the solid product dried, with the exclusion of oxygen, at temperatures above 200°C.

2. A process as claimed in Claim 1, wherein an inert gas is injected into the suspension in the vertical chamber.

3. A process as claimed in Claim 1 or 2, wherein the solid products are subjected to a subsequent de-acidification.

4. A process as claimed in Claims 1 to 3, wherein the suspended particles are heated to a temperature of from 400 to 600°C.

5. A process as claimed in any of Claims 1 to 4, wherein silicon tetrachloride or preferably an alkyl- or an aryl- or an alkyl-aryl-halogensilane or a silane ester are used as substance (A).

6. A process as claimed in any of Claims 1 to 5, wherein the oxides are reacted initially with an alcoholic alkali.

7. A process as claimed in Claim 1 wherein the treatment is effected in a treatment chamber which has been so dimensioned that the suspension moves steadily upwards and continuously leaves the upper part of the chamber, preferably after traversing a settling zone, while the waste gases are also evacuated at the upper end of the reaction chamber.

8. A process as claimed in any of Claims 1 to 7, wherein the vertical chamber is internally heated.

9. A process as claimed in any of Claims 1 to 8, wherein the heat required is supplied partly by steam and/or a hot inert gas.

10. A process as claimed in any of Claims 1 to 9, wherein the heat and the steam required for the treatment are generated by burning an oxyhydrogen flame.

11. A process as claimed in any of Claims 2 to 10, wherein the steam and/or the inert gas are employed for the pneumatic conveying of the reactants.

12. A process for the surface treatment of highly dispersed oxides as claimed in Claim 1, substantially as described with reference to any of the Examples and to the accompanying drawings.

13. Treated oxides obtained by a process as claimed in any of the preceding claims.

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